

# PCT

# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>4</sup> :		(11) International Publication Number: WO 89	)/12407
A23L 3/38, A23C 9/123	A1	(43) International Publication Date: 28 December 1989 (	(28.12.89)
(21) International Application Number: PCT/II (22) International Filing Date: 20 June 1989 (30) Priority data: 48112 A/88 20 June 1988 (20.06.88)  71) Applicant (for all designated States excelling Innovative Freeze-Drying Pro((IFDP) N.V. [NL/NL]; Deruyterkade 58A, 837, Willemstadt, Curacao (AN). (72) Inventors; and (75) Inventors/Applicants (for US only): COSTANZO, IT]; CALCAVECCHIA, Biagio [IT/IT]; Via Agona, 37, 1-95126 Catania (IT). (74) Agents: IANNONE, Carlo, Luigi et al.; Ing. B Zanardo Roma S.p.A., Via Piemonte, 26, I-001 (IT).	(20.06.	(European patent), BF (OAPI patent), BG, B patent), BR, CF (OAPI patent), CG (OAPI patent), CH (European patent), DK, FI, FR (European patent), COAPI patent), (European patent), BB, GB (European patent), HU ropean patent), JP, KP, KR, LK, LU, LU (European patent), MC, MG, ML (OAPI patent), MR (OAPI MW, NL, NL (European patent), NO, RO, SE (European patent), SN (OAPI patent), TG (OAPI patent), US.  Published  With international search report.  Before the expiration of the time limit for americal claims and to be republished in the event of the amendments.	sj (OAPI ent), CH, DE, DE, tent), GA J, IT (Eu- ppean pa- I patent), J, SE, SE D (OAPI ending the

(54) Title: A PROCESS FOR PREPARING LYOPHILIZED FOODSTUFFS IN THE SOLID STATE AND HAVING PREFERABLY PREDETERMINED GEOMETRICAL SHAPES, OF HIGH NUTRITIONAL VALUE AND READY TO USE, AND THE PRODUCTS SO OBTAINED

### (57) Abstract

This invention relates to a process for the production of lyophilized foodstuffs in solid form and based preferably on fresh foodstuffs such as in particular yoghurt, milk, fruit and so on, said process comprising the steps of: a) mixing the ingredients; b) homogenizing said ingredients or distributing the same uniformly throughout the mixture, possibly with pasteurization; b1) trapping air into the mixture obtained in steps a) and b) until reaching a percentage between 3 and 90 % of the initial volume of the ingredients, preferably between 10 and 30 %, and b2) cooling said mixture with continuous mixing, simultaneously or successively, until reaching a temperature between -5°C and -12°C, preferably between -5°C and -9°C; c) shaping the product obtained in steps b) or b1) and b2) into the desired shape, preferably by extrusion; d) deep-freezing the product obtained in step c), at a temperature between -15 and -60°C, and preferably between -30°C and -40°C; e) lyophilizing the product employing lyophilization parameters suitable to the type and sizes of the product to be processed, until reaching a residual moisture between 0 and 10 %, and preferably between 2 and 6 % (determined by the Karl Fisher method); the invention also relating to the products obtained by the process mentioned above.



# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



# INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

WO 89/12407 (11) International Publication Number: (51) International Patent Classification 4: **A1** 28 December 1989 (28.12.89) A23L 3/38, A23C 9/123 (43) International Publication Date: (81) Designated States: AT, AT (European patent), AU, BB, BE (European patent), BF (OAPI patent), BG, BJ (OAPI patent), BR, CF (OAPI patent), CG (OAPI patent), CH, CH (European patent), CM (OAPI patent), DE, DE (European patent), DK, FI, FR (European patent), GA (OAPI patent), GB, GB (European patent), HU, IT (European patent), JP, KP, KR, LK, LU, LU (European patent), MC, MG, ML (OAPI patent), MR (OAPI patent), MW, NL, NL (European patent), NO, RO, SD, SE, SE (European patent), SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI patent), US. PCT/IT89/00044 (21) International Application Number: 20 June 1989 (20.06.89) (22) International Filing Date: (30) Priority data: 20 June 1988 (20.06.88) IT 48112 A/88 (71) Applicant (for all designated States except US): LIOPRO-DOTTI S.R.L. [IT/IT]; Via Laurentina Km 27, I-00040 Pomezia Roma (IT). **Published** (72) Inventors; and
(75) Inventors/Applicants (for US only): COSTANZO, Luigi [IT/IT]; CALCAVECCHIA, Biagio [IT/IT]; Via Artale Alagona, 37, I-95126 Catania (IT). With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments. (74) Agents: IANNONE, Carlo, Luigi et al.; Ing. Barzanò & Zanardo Roma S.p.A., Via Piemonte, 26, I-00187 Rome

(54) Title: A PROCESS FOR PREPARING LYOPHILIZED FOODSTUFFS IN THE SOLID STATE AND HAVING PREFERABLY PREDETERMINED GEOMETRICAL SHAPES, OF HIGH NUTRITIONAL VALUE AND READY TO USE, AND THE PRODUCTS SO OBTAINED

#### (57) Abstract

This invention relates to a process for the production of lyophilized foodstuffs in solid form and based preferably on fresh foodstuffs such as in particular yoghurt, milk, fruit and so on, said process comprising the steps of: a) mixing the ingredients; b) homogenizing said ingredients or distributing the same uniformly throughout the mixture, possibly with pasteurization; b1) trapping air into the mixture obtained in steps a) and b) until reaching a percentage between 3 and 90 % of the initial volume of the ingredients, preferably between 10 and 30 %, and b2) cooling said mixture with continuous mixing, simultaneously or successively, until reaching a temperature between -5°C and -12°C, preferably between -5°C and -9°C; c) shaping the product obtained in steps b) or b1) and b2) into the desired shape, preferably by extrusion; d) deep-freezing the product obtained in step c), at a temperature between -15 and -60°C, and preferably between -30°C and -40°C; e) lyophilizing the product employing lyophilization parameters suitable to the type and sizes of the product to be processed, until reaching a residual moisture between 0 and 10 %, and preferably between 2 and 6 % (determined by the Karl Fisher method); the invention also relating to the products obtained by the process mentioned above.

A PROCESS FOR PREPARING LYOPHILIZED FOODSTUFFS IN THE SOLID STATE AND HAVING PREFERABLY PREDETERMINED GEOMETRICAL SHAPES, OF HIGH NUTRITIONAL VALUE AND READY TO USE, AND THE PRODUCTS SO OBTAINED

5

This invention relates to a process allowing lyophilized foodstuffs to be produced having preferably predetermined geometrical shapes and ready to use, as well as of high nutritional value, and to the products so obtained.

More particularly, this invention relates to a preparation procedure whose steps allow lyophilized products to be obtained having predetermined geometrical shape like those claimed herein.

15

10

Moreover, this invention relates to products of the type mentioned above which, though in the solid form and ready to be eaten, and accordingly having all advantageous features of such conformations, keep substantially unaltered the original nutritional features of the ingredients employed in the formulation itself.

20

The use of snacks ready to eat is very widespread because of the remarkable advantages in their commercial distribution and in eating the same.

25

Particular reference is made here to their easy stockage and storage, as well as to the practical advantages on the user's part in transporting and eat-ing them.

30

Foodstuffs like yoghurt, fruit-flavoured yoghurt or fruit- or milk-based foodstuffs are in many cases preferred because they are considered as richer in nutritional values and as healthier.

The main drawbacks of such kind of foodstuffs are those stemming from their short conservation time even at low temperatures, unless they are stabilized by heat treating.

35

10

15

ر 20

25

30

35

Such ways of conservation in the first instance limit their employment, while in the second case they give rise to a more or less remarkable reduction in the amounts of nutritional stuffs contained in the product.

In particular, for instance, in the case of a trip to the seaside it is necessary to have a refrigerated bag or a refrigerator in order to have the possibility of eating a yoghurt or a fresh fruit smash, so that in most cases the use of such foodstuffs is to be renounced.

Accordingly, the Applicant has devised to realize lyophilized foodstuffs in the solid form and ready to eat, having all conservation and transportation practical requisites that are characteristic of such type of products, such foodstuffs also having all features and nutritional values of foodstuffs like yoghurt, milk, fruit, meat, eggs or vegetables.

Thus, a process has been realized in the laboratories of the Applicant for the production of foodstuffs having the characteristics mentioned above, said process comprising procedural steps that allow lyophilized foodstuffs to be obtained in predetermined geometrical shape and ready to use, said foodstuffs keeping substantially unaltered all organoleptic and nutritional features of the ingredients employed in their formulation.

Such results are obtained according to the present invention through the realization of a process wherein, after mixing all liquid, liquid and solid (dusts and/or pellets) ingredients designed, a step is carried out in which air is trapped inside the product with simultaneous or separate cooling, under continuous stirring, said step giving the mixture a plastic state of high viscosity that allows the next shaping operation to be performed, preferably by extrusion. Then the product is deep-frozen and then lyophilized so that it is obtained in the solid form.

10

15

20

25

35

The advantages stemming from the realization of products like those obtained by means of the process according to the present invention are self-evident and in particular they can be considered as commercial-type advantages and as consumer's advantages.

Among the commercial-type advantages, the reduced weight of the product can be included, so that it can be more easily manipulated and transported, and the possibility of storing the same at room temperature, so that a remarkable reduction in distribution costs is obtained.

The consumer will have at his/her disposal a product that can be more easily transported with respect to the similar non-lyophilized product, and in addition he/she will have the possibility of carrying the product without necessarily having at disposal a refrigerator of similar cooling means.

Accordingly, it is a specific object of the present invention a process for the preparation of lyophilized foodstuffs having geometrical shapes and ready to use, of high nutritional value, and based preferably on fresh foods, such as in particular yoghurt, milk, fruit, said process comprising the steps of:

- a) mixing the ingredients;
- b) homogenizing said ingredients or distributing them uniformly throughout the mixture;
  - c) forming the product so obtained into the desired shape;
- d) deep-freezing the product at a temperature between
   30 -15°C and -60°C;
   and
  - e) lyophilizing the product with lyophilization parameters suitable to the type and sizes of the product under treatment, till obtaining a residual moisture between 0 and 10 % (determined by the Karl Fisher method).

    According to a preferred embodiment of the process of

10

15

20

25

30

35

the present invention, after said step b) a step bl) can be suitably carried out for trapping air into the mixture at a percentage between 3 and 90 % with respect to the initial volume of the ingredients.

Moreover, a step b2) can also be included in the process of cooling, under continuous mixing, the mixture until reaching a temperature in the range from -5 to - 12°C which step can be performed independently of the fact that said step b1) is carried out.

When both steps b1) and b2) are carried out, according to the present invention the process in question can also be realized by continuous mixing, so trapping air, while cooling the mixture, or trapping air into the mixture and cooling and mixing continuously, simultaneously or successively.

Again according to the present invention, step b) can be completed with a pasteurization of the mixture.

Preferably the product is deep-frozen in step d) at a temperature from -30°C to -40°C, and lyophilization according to step e) is performed till a residual moisture is obtained in the range from 2 to 6% (determined by the Karl Fisher method).

In step b1) in which air is trapped, preferably air will be trapped into the mixture till a percentage between 10 and 30 % of the initial volume of the ingredients and the cooling step b2) is carried out until a temperature of the product is obtained between -5 and -9°C.

Next to step e) the product obtained can be iced with chocolate and its substitutes or similar products, or it can be coated with a film of cellulose, gum, wax, maltodextrins, milk proteins, sugars and so on.

As already mentioned above, the ingredients treated by the process according to the present invention can be all food products and food additives allowed by the laws, but fresh foodstuffs having a good nutritional

10

15

20 🐔

25

30

35

value that require low temperature conservation, like yoghurt, milk, fruit etc. are to be preferred.

If a sugar product is to be employed in the formulation, it is preferable to use dextrose because it is scarcely hygroscopic and in addition has a negative heat of solution. These features make the lyophilized product more capable to resist the action of the environmental humidity when the package containing the product is opened, and because it is more palatable as it gives a pleasant feeling of freshness on dissolving in the mouth.

In case both liquid and solid ingredients are present, step a) is preferably performed:

- al) by mixing the solid ingredients;
- a2) by mixing the liquid ingredients;
- a3) by mixing both ingredients al) and a2).

Step b) can be performed with or without cooling. Preferably such step will be carried out with cooling in order to warrant a better conservation of the nutritional and organoleptic qualities of the raw materials employed and to avoid any possible microbial proliferations.

The trapping of air into the mixture provided in step bl) can be carried out in a spontaneous way operating with open systems or with closed systems, injecting air under pressure, or finally by means of other procedures capable of realizing such operation together with the two others or before them.

However it can be remarked that when step bl) is performed in closed systems with mixtures prepared according to the step b) and containing living milk ferments, the survival of said ferments depends on the value of pressure inside the system.

Indeed, it has been observed that survival of milk ferments decreases strongly with increasing the working pressure. Accordingly, in order to obtain the maximum

10

15

20

25

30

35

possible survival of living milk ferments, the operation is to be preferably carried out at atmospheric pressure or anyway so as not to reach pressures above the relative pressure of 1 bar.

Though there are many problems in performing correctly the operation of trapping air into the mixture, such operation is very important because it yields a set of economical advantages such as:

- speeding up the lyophilization process, thanks to the formation of empty spaces in the mixture mass that favour the going out of water vapour;
- the possibility of lyophilizing mixtures with remarkably high dry residue because the possibility of melting caused by high partial pressures due to mass transfer difficulties is minimized.

Moreover, the three operations of steps b1) and b2) as a whole and their correct realization give further advantages such as:

- the formation of water crystallization nuclei of very small sizes;
  - the instantaneous reconstitution;
- the finely pitted structure and the crumbly consistency, which is a peculiar characteristic of the products obtained with the process of the present invention.

Step c) consisting in the operation of shaping the product which has been made viscous and plastic by the preceding cooling with continuous mixing, can be realized according to the present invention preferably by extrusion and drawing, or by filling moulds.

As regards the lyophilization step f), it will be carried out under highly decreased pressure.

The lyophilized product obtained according to the process disclosed above has a consistency or texture which is characteristic and can be described by determining the following properties of the product:

- 1) resistance to penetration;
- crumblyness;
- 3) work to be done for a 6 mm penetration into the product mass.

Such properties have been determined employing a 4301 Instron dynamometer according to the following procedures:

Test 1: penetration resistance Materials and method:

- chisel-type bit, obtained from a steel cylinder of 10 8 mm diameter, having the following characteristics:

o mm diameter, maring	
- cutting base length	0,5 mm
- cutting base width	8 mm
	220
- angle of the tapered part	
- feed speed	50 mm/min
- applied force	10 kg

#### Estimate:

From the records of the resistance of the product against the penetration of the tool, a curve is obtained whose initial part can be likened to a straight line. The slope of such straight portion of the curve represents the value of the penetration resistance expressed as kg/mm. The values of the measurements are obtained from the average of at least 10 tests on samples of identical sizes.

Test 2: crumblyness and work done for 6 mm penenetration depth

Material and method

- cylindrical bit of diameter	U.8 mm
- feed speed	2 mm/min
- applied force	10 kg
- penetration depth	6 mm

## Estimate:

)

By performing the test as above, a curve with two peaks is obtained.

The height of the first peak represents the crumblyness that is expressed as kg. The second determinable property is the

10

35

additives

work required for penetrating 6 mm into the product, said work being proportional to the area under the curve and being expressed as kg x mm.

The average properties determined in the lyophilized products obtained according to the present process are as follows:

- 1) penetration resistance: from 0.1 to 10 kg/mm, preferably from 2 to 5 kg/mm
- 2) crumblyness: from 0.05 to 5 kg, preferably from 0.1 to 3 kg
- 3) work for penetrating a 6 mm depth: from 0.05 to 10 kg x mm, preferably from 1 to 8.5 kg x mm.

Moreover, a product is to be also considered as comprised in the scope of the present invention when it is obtained by the same process as above and comprises:

fresh yoghurt and/or fresh milk

fruit

other liquid or solid ingredients

0-100 %

0-100 %

More particularly, the ingredients that can be present are: fresh milk, 9-80 %; fruit, 2-90 %; other liquid or solid ingredients, 0-70 %, and additives, 0-10%.

In case yoghurt is present, it is in amounts between 25 10 and 100 %, and preferably it will be present at a percentage of at least 70 %.

Such other liquid or solid ingredients mentioned above will consist of meat, vegetables, eggs, sugars, maltodextrins, glucose syrups.

The additives can be thickening agents, emulsifiers, colouring agents, flavours, anti-oxidizing agents, acidifiers.

Fruit employed can be fresh fruit, or deep-frozen fruit, dried or pasteurized fruit, such fruit being in the form of pellets or of a smash having a natural or strengthened concentration.

Some practical examples of realization of the process

70 0 %

10

30

35

according to the present invention will be reported in the following just for illustrative and not for limitative purposes.

#### EXAMPLE I

5	The	following	ingredients	are	subjected	to	the	pro-
	cess:							

- natural whole yoghurt	10.0 %
- strawberry smash	13.1 %
- dried red beets	1.2 %
- carrageenins	0.1 %
- strawberry flavour	0.2 %
- soya lecithin	0.1 %
- dextrose	15.2 %
- L-ascorbic acid	0.1 %

15 - coating: based on hydrogenated, lemon-lavoured vegetable fats.

First the dried red beets, carrageenins, soya lecithin, dextrose and ascorbic acid (solid ingredients) are mixed, and the mixture is then added together with

strawberry flavour to the yog urth and to the strawberry smash.

The mass is mixed further so as to make the distribution of the ingredients uniform throughout the mixture, keeping the temperature at  $+4\,^{\circ}\text{C}$ .

25 Then the mixture is cooled with continuous mixing and under atmospheric pressure till the product starts showing a semisolid texture (between -5°C and -12°C) and an air percentage of 10-30 % of the initial volume has been trapped.

Then the product is subjected to extrusion forming, taking care that the product does not undergo superficial thawing and that trapped air does not leak out.

The product is deep-frozen at a temperature between  $-30^{\circ}$ C and  $-40^{\circ}$ C, and next it is placed on the previously cooled trays of the lyostat at the ratio of 6.8 kg/m<sup>2</sup>, keeping the temperature of said product constantly at

the value of -30°C.

Lyophilization in the case of product rods of 1.2 cm diameter is carried out as follows:

- maximum temperature of plates 65°C
- 5 maximum temperature of the product surface 30°C

0 2 --- 1

- pressure inside the lyostat 0.3 mm Hg
When the lyophilization process is over, the residual moisture is preferably between 4 and 6 %.

The value of consistency or texture determined on the Instron apparatus and following the procedures already disclosed above, is as follows:

- penetration resistance from 0.5 to 2.5 kg/mm - crumblyness from 0.3 to 0.5 kg

15 - work done in penetrating a

6 mm depth from 1.5 to 2.2 kgxmm

The lyophilized product is taken away from the vessels, subjected to selection, iced with lemon-flavoured coating and packaged.

It is also possible to obtain a product having more flavours combined together within the same piece, if mixtures having similar features but different flavours are processed in a parallel way, extruding the mixture with an extruder capable of putting together the mixtures. For instance, it is possible to extrude a small bar a half of which has a "strawberry yoghurt" flavour while the other half has a "vanillin yoghurt" flavour.

Vanillin yoghurt is prepared in that case in the same way as the strawberry yoghurt, employing the following

30 formulation:

- natural whole yoghurt		80	.0 %
	- dextrose	19	.6 %
	- carrageenins	0	.1 %
	- soya lecithin	0	.1 %
35	- ascorbic acid	. 0	.1 %
	- vanillin flavour	0	.1 %

20

#### - 11 -

#### EXAMPLE II

The following ingredients are processed:

	- apple smash	35.0 %	
	- dextrose	12.0 %	
5	- lemon juice	2.4 %	
	- pectin	0.5 %	
	- natural flavours	0.1 %	

- deep-frozen apple dice i.q.f. (individual quick frozen)
- coating: melting chocolate containing 32-34 cocoa butter.

All solid ingredients, i.e. dextrose and pectin, are first mixed.

Then lemon juice, flavours and the solid ingredient mixture mentioned above are added to the apple smash kept under stirring.

The whole mixture is homogenized and pasteurized.

The mixture is cooled with continuous mixing till reaching a temperature in the range from -5 to -12°C after blowing air in amounts from 10 to 30 % of the initial volume of the product.

The apple dice are then added to the cold and viscous mass previously obtained, dispersing them throughout the same in a uniform way.

25 The product is then subjected to the forming operation for obtaining the predetermined sizes.

The product is deep-frozen at a temperature between -30°C and -40°C, and next it is placed on the pre-cooled trays of the lyostat.

Finally the product is lyophilized by means of a cycle suitable to the sizes of the pieces as well as to the amounts of them on the trays. In particular, in the case of 1 cm thickness bars, lyophilization is carried out as follows:

- maximum temperature of plates 60 °C - maximum temperature of the product surface 25 °C

10

15

30

- pressure inside the lyostat 0.3 mmHg When the lyophilization process is over, the residual moisture of the product is 3%.

The value of consistency determined on the Instrom apparatus and following the procedure already disclosed above, is as follows:

- penetration resistance

from 0.2 to 2 kg/mm

- crumblyness

from 0.1 to 0.3 kg

- work for a penetration depth

of 6 mm

from 0.7 to 3.2 kg x mm

The lyophilized product is unloaded from the trays, subjected to selection for removing broken pieces, and sent to the chocolate-icing plant.

Finally the product is again subjected to selection and packaged.

As a variant to the procedure detailed above, during the icing operation the product can be spread with dry or dried fruit, or with puffed cereals, small chocolate pieces and so on.

# EXAMPLE III

20 The following ingredients are processed:

- coco smash

74.9 %

- dextrose

25.0 %

- natural flavours

0.1 %

- coating: melting chocolate containing 32-34 % cocoa

25 butter.

Coco smash, dextrose and flavours are all mixed with care.

The mixture so obtained is then cooled with continuous mixing until it acquires a semisolid texture (temperature between -5 and -12°C) and has trapped air in amounts between 10 and 30 % of the initial volume of the product.

The viscous mass so obtained is then extruded into the desired shape.

The product so formed is deep-frozen at a temperature

20

25

between -30°C and -40°C and then placed on the precooled trays of the lyostat.

Finally the product is lyophilized by means of a cycle fit for the piece sizes and the amounts of the products arranged on the trays. •

In particular, when processing small rods of 1 x 1 x 4 cm sizes and distributing the pieces on the trays at a density of 6 kg/m<sup>2</sup>, the lyophilization is carried out as follows:

- maximum temperature of plates 60°C
- maximum temperature on the surface
of the product 25°C
- pressure inside the lyostat 0.3 mm Hg

When the lyophilization is over, the product has a

residual moisture between 4 % and 6 %.

The consistency or texture determined on the Instant apparatus employing the procedures disclosed above is as follows:

- penetration resistance from 0.3 to 2.5 kg/mm - crumblyness from 0.1 to 0.8 kg

- work for a penetration

depth of 6 mm from 0.7 to 4 kg x mm

The lyophilized product is unloaded from the trays subjected to selection for removing broken pieces and sent to the chocolate-icing plant.

Finally the product is definitively selected and packaged.

#### EXAMPLE IV

The following ingredients are processed:

30	- whole fresh milk	45.0	%
30	- strawberry smash	32.0	7.
	- dextrose	15.3	or A
	- fresh liquid cream	2.0	# /s
	milk proteins	2.0	7.
35	- natural vegetable extract	3.5	Z

from 1 to 3 kg/mm

from 0.4 to 0.8 kg

5

15

20

25

30

35

	-14 -		PC1/1189/0	J0044
+ L-ascorbic	acid		0.1	 •
- natural fla			0.1	
- coating:		drocenated.	10.0	
flavoured fat		21066	AFRETADIC	A G 11 T T T I I
	ingredients	ara firet :	mivade dave	rono =:31
proteins and				
are separate			=	-
tracts and na	- ·		maturar veg	Erapie ex
	mixtures ar		ed to the	ctrauharra
BOCH CHE	mixtures ar	e then add	ed to the	attomactt;
smash which h	ad been previ	iously thaw	red and kept	t at +4°C.
The whole	mixture is	then homo	genized and	pasteur.
ized.				
The mixtur	e is then cod	oled with o	continuous	mixing un-
til the produ	ict starts s	howing a s	emisolid co	onsistency
or texture s				
(from -5°C to				n amousts
from 10 to 30				
_	roduct is sul			
•	t so obtaine	_		
ture between				
viously coole		the lyost	at, keeping	the ten-
perature at -			4	
• •	ation is car			•
lowing parame			product in	the shape
of rods of 1.				0.0
- maximum tem	-		70	
- maximum tem	perature of	the product		5°C
surface .				-
- pressure in	•			.3 mm Hg
	ial moisture			
	stency or tex	•		
on the Insti	on apparatus	- with the	= broceance	s alleady

disclosed above is as follows:

- penetration resistance

- crumblyness

	- work for 6 mm penetration
	depth . from 2.5 to 3.5 kg x mm
	The lyophilized product is subjected to selection,
	then iced with the vanillin-flavoured coating and pack-
5	aged.
	EXAMPLE V
	The product is prepared employing the following for-
	mulation:
	- skimmed milk 71.5 %
10	- dextrose 17.8 %
	- egg yolk 10.7 %
	Dextrose is added to yolks and the whole is speedily
	mixed until complete dissolution. Then, milk is added.
	The mixture is again mixed speedily for making it
15	homogeneous and then it is pasteurized.
	Air is injected in amounts from 10 to 30 % of the
	initial volume of the mixture, and the whole mass is
	cooled with continuous mixing until reaching a tempera-
	ture between -5 and -12°C.
20 💰	The mixture which is semisolid at this point is ex-
•	truded into the predetermined shape.
	The product is deep-frozen at a temperature between
	-30°C and -40°C and placed on the trays of the
	lyostat.
25	Lyophilization is then performed, which in the case
	of 1.2 cm diameter rods distributed on the trays at a
	density of $6.2 \text{ kg/m}^2$ is carried out according to the
	following parameters:
	- maximum temperature of plates 65°C
<b>3</b> 0	- maximum temperature of the product
	surface 35°C

sidual moisture between 2 and 6 %. The consistency or texture determined on the Instron 35

Lyophilization is carried out so as to obtain a re-

0.3 mm Hg

- pressure inside the lyostat

with the procedures already	TOI ES EL SVOCE DEMOLINSM
lows:	
- penetration resistance	from $0.8$ to $2.7$ kg/mm
- crumblyness	from 0.1 to 0.7 kg
- work for 6 mm penetration	
depth	from 1.4 to 2.6 kg x mm
The product is selected	and packaged with a high-
barrier material.	
EXAMPL	E VI
_	

The product is prepared employing the following formulation:

	- dextrose	6.3 %	
	- pectin	0.1 %	
	- pasteurized whole milk	9.5 %	
15	- water	41.0 %	
	- glucose syrup	6.3 %	
	- cooked minced meat	7.8 %	
	- hydrogenated vegetable fat	3.1 %	
,	- maltodextrins	6.3 %	
20	- cocoa	0.4 %	
	- soya lecithin	0.3 %	
	- salt	0.1 %	
	- natural flavours	0.1 %	
	- whole eggs	6.3 %	
25	- natural orange juice	8.6 %	
	- mixed dried fruit in the form		
	of dice	2.4 %	
	- raisin	1.4 %	
•	- [415111		

- coating: milk chocolate

Powder ingredients are mixed (dextrose, pectin, maltodextrins, cocoa, soya lecithin, salt).

The liquid ingredients are mixed (milk, water, glucose syrup, natural flavours, eggs, orange juice), fats previously molten, and meat.

35 / While such liquid ingredients are stirred, the mix-

10

20

30

ture of the solid ingredients is added.

The whole mixture is homogenized and pasteurized.

The mixture so obtained is added with air through injection (from 10 to 30 % of the initial volume) and then it is cooled with continuous mixing until it becomes a semisolid mass (at a temperature between -5°C -12°C).

Dice of dried fruit and raisin are added, distributing the same uniformly throughout the whole mass.

The product is given the shape of a small bar, said product having a viscous but plastic consistency, employing some moulds and operating in a suitable environment so as to avoid any possible increase in surface temperature.

15 The product is deep-frozen until reaching a temperor ture between -30°C and -40°C.

> The product is taken out of the moulds and placed on trays at the density of 7.6 kg/m<sup>2</sup> operating in a cold environment in order to prevent the product from thawing.

> The product is then lyophilized employing in the specific case of bars of 1 cm thickness the following parameters:

80°C - maximum temperature of plates

25 - maximum temperature of the product

45°C surface

0.3 mm Hg - pressure inside the lyostat The final moisture is in the range from 3 to 6 %.

The consistency of the product determined on the Instron apparatus by the procedures mentioned above is as follows:

- penetration resistance

from 0.4 to 2 kg/mm

- crumblyness

from 0.05 to 0.7 kg

from 0.4 to 3.5 kg x - work for 6 mm penetration depth

35 · The small bars lyophilized are selected and they can

be optionally spread with small grains of dried or roasted fruit, dry fruit, puffed cereals, chocolate flakes, and so on, and/or coated with milk chocolate.

Finally the product is packaged.

This invention has been disclosed according to some preferred embodiments of the same, but it is to be understood that modifications and/or changes can be introduced in the same by those who are skilled in the art without departing from the spirit and scope of the invention for which a priority right is claimed.

10

15

20

25

30

#### CLAIMS

- 1. A process for the preparation of lyophilized foodstuffs having preferably predetermined geometrical shapes and ready to eat, said process being characterized in that it comprises the steps of:
  - a) mixing the ingredients;
- b) homogenizing said ingredients or distributing the same uniformly throughout the mixture;
- c) shaping the product so obtained into the desired shape;
- d) deep-freezing the product so formed at a temperature between -15°C and -60°C; and
- e) lyophilizing the product so obtained, with lyophilization parameters so as to obtain a residual moisture between 0 and 10 % (determined by the Karl Fisher method).
- 2. A process according to claim 1, characterized that after step e) an icing step is carried out  $\exp(-i\lambda)$ ing chocolate and its substitutes or similar products, or a coating step is performed for obtaining a coating consisting of a film of cellulose, or gums, or waxes or maltodextrins or milk proteins or sugars.
- 3. A process according to claims 1 or 2, characterized in that after step b) a step b1) is performed for trapping air into the mixture up to a percentage between 3 and 90 % of the initial volume of the ingredients.
- 4. A process according to claims 1 or 2, characterized in that after step b) a step b2) is performed of cooling under continuous mixing the mixture until reaching a temperature between -5 and -12°C.
- 5. A process according to claims 1 or 2, characterized in that, after step b), a step bl) for trapping air into the mixture up to a percentage between 3 and 90 %of the initial volume of the ingredients, and a step b2) of cooling undercontinuous mixing until reaching a tem-35

15

20

25

perature between -5 and -12°C, are carried out.

- 6. A process according to claim 5, characterized in that said steps bl) and b2) are carried out by continuous mixing, trapping air and then cooling the mixture.
- 7. A process according to claim 5, characterized in that said steps b1) and b2) are carried out by trapping air into the mixture, cooling and mixing continuously, simultaneously with the operation of air trapping or after that.
- 8. A process according to claims 3 or 5, or 6 or 7, wherein said step b1) is carried out by trapping air up to a percentage between 10 and 30 % of the initial volume of the ingredients.
  - 9. A process according to claims 3 or 5 or 6 or 7 or 8, wherein said air trapping into the mixture is performed operating spontaneously in open systems.
  - 10. A process according to claims 3 or 5 or 6 or 7 or 8, wherein said air trapping into the mixture is performed by injecting air under pressure in closed systems.
  - 11. A process according to any one of the claims 3-10 wherein said steps b1) and/or b2) are carried out under a relative pressure equal to or less than 1 bar.
  - 12. A process according to claims 4 or 5 or 6 or 7, wherein said step b2) is performed by cooling the product until reaching a temperature between -5°C and -9°C.
  - 13. A process according to any one of the preceding claims, characterized in that a pasteurization of the product is performed in said step b).
- 14. A process according to anyone of the preceding claims, characterized in that said product is deepfrozen according to step d) at a temperature in the range between -30°C and -40°C.
- 15. A process according to any one of the preceding claims, characterized in that the lyophilization according to said step e) is carried out until reaching a re-

20

30

sidual moisture between 2 and 6 % (determined according to the procedure by Karl Fisher).

- 16. A process according to any one of the preceding claims, wherein the step a) in the presence of liquid and solid ingredients is carried out by:
  - al) mixing the solid ingredients
  - a2) mixing the liquid ingredients
  - a3) mixing the ingredients of al) and a2).
- 17. A process according to any one of the preceding claims, wherein said step b) is carried out with refrigeration.
  - 18. A process according to any one of the preceding claims, wherein said step c) of shaping the product is realized by filling moulds.
- 19. A process according to any one of the preceding claims 4-17, wherein said shaping step c) is realized extrusion and/or drawing.
  - 20. A process according to any one of the preceding claims, wherein said lyophilization step e) is realized under strongly decreased pressure.
  - 21. Lyophilized foodstuffs having geometrical shapes and obtained by the process according to any one of the preceding claims, said foodstuffs being characterized in that they are ready to eat and comprise:
- 25 fresh yoghurt and/or fresh milk 0-100 % fruit 0-100 % other liquid or solid ingredients 0-100 % additives 0-10 %
  - 22. A product according to claim 21 characterized in that it comprises:

fresh milk 9-80 % fruit 2-90 % other liquid or solid ingredients 0-70 % additives 0-10 %

23. A process according to claim 21, characterized in that it comprises yoghurt between 10 and 100 %, and ad-

10

ditives between 0 and 10 %, and other liquid or solid ingredients between 0 and 30 %, as a whole.

24. A product according to claim 23, wherein yoghurt is present in amounts of at least 70 %.

25. A product according to claim 21, characterized in that it comprises:

fresh	yoghurt			10-80	:
fruit			•	20-90	. 3
other	liquid or	solid	ingredients	0-70	~
addit	ives			0-10	"

26. A product according to claim 25, wherein yearurt and fruit are present as a whole in amounts of 70-100 %.

27. A product according to any one of the preceding claims 21-26, wherein said other liquid or solid ingredients consist of meat, eggs, vegetables, sugars, maltoder ins, glucose syrups.

- 28. A product according to any one of the preceding claims, wherein said additives consist of thickening agents, emulsifiers, colouring agents, flavouring agents, anti-oxidizing agents, acidifying agents.
- 29. A product according to any one of the preceding claims 21, 22, 25, 26 wherein said fruit is fresh fruit, or it is deep-frozen, or dried, or pasteurized fruit, in the form of pellets of a smash at the natural or a strengthened concentration.
- 30. A product according to any one of the preceding claims 21-28, whose consistency determined by the method described or texture is identified by the following values:

-	penetration	resistance	٠.		0.1 -	10	kg/mm
-	crumblyness		•	•	0.05 -	5	kg

- work done for a 6 mm penetration

30 depth into the product 0.5 -10 kg x mm

31. A product according to claim 30, whose consistency or texture is identified by the following values:

- penetration resistance

2 - 5 kg/mm

- crumblyness

0,1 - 3 kg

5 - work done for a 6 mm penetration depth into the product

1 - 8,5 kg x mm

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/IT 89/00044

III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Category* Citation of Document, " with indication, where appropriate, of the relevant passages "  X FR, A, 1600708 (SICALY) 4 September 1970 see page 2, paragraph 4; page 3, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON) 26 June 1956 see figures 1-3; claims 1,8  A 23 L 3/38, A 23 C 9/123  III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Category* Citation of Document, " with indication, where appropriate, of the relevant passages " Relevant to Claim 18,20  X FR, A, 1600708 (SICALY) 4 September 1970 see page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON) 26 June 1956 see figures 1-3; claims 1,8  DE, A, 1692705 (P. KÖLLN)						
III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Classification System   Classification Symbols  III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Category*   Citation of Document, "I with indication, where appropriate of the relevant passages 12  X FR, A, 1600708 (SICALY)  4 September 1970  5 See page 2, lines 11-45; abstract, points 1-3  X DE, A, 1816761 (UNILEVER)  24 July 1969  5 See page 2, paragraph 4; page 3, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON)  26 June 1956  5 See figures 1-3; claims 1,8						
III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Classification System   Classification Symbols  III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Calegory*   Citation of Document, " with indication, where appropriate, of the relevant passages 12   Relevant to Claim  X   FR, A, 1600708 (SICALY)   1,2,14   18,20    X   DE, A, 1816761 (UNILEVER)   24 July 1969   see page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X   US, A, 2751687 (R.F. COLTON)   26 June 1956   see figures 1-3; claims 1,8						
Classification System   Classification Symbols   Classification Symbols   Classification Symbols   Classification Symbols						
Classification System   Classification Symbols  TPC 4 A 23 L, A 23 C  Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched*  III. DOCUMENTS CONSIDERED TO BE RELEVANT*  Category* Citation of Document, "I with indication, where appropriate, of the relevant passages "I Relevant to Claim  X FR, A, 1600708 (SICALY)  4 September 1970  5 see page 2, lines 11-45; abstract, points 1-3  X DE, A, 1816761 (UNILEVER)  24 July 1969  5 see page 2, paragraph 4; page 3, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON)  26 June 1956  5 see figures 1-3; claims 1,8						
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched.  III. DOCUMENTS CONSIDERED TO BE RELEVANT.  Category.* Citation of Document, 11 with indication, where appropriate, of the relevant passages 12 Relevant to Claim  X FR, A, 1600708 (SICALY)  4 September 1970  5ee page 2, lines 11-45; abstract, points 1-3  X DE, A, 1816761 (UNILEVER)  24 July 1969  5ee page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON)  26 June 1956  5ee figures 1-3; claims 1,8						
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched.  III. DOCUMENTS CONSIDERED TO BE RELEVANT.  Category. Citation of Document, "I with indication, where appropriate, of the relevant passages 12 Relevant to Claim  X FR, A, 1600708 (SICALY) 4 September 1970 5ee page 2, lines 11-45; abstract, points 1-3  X DE, A, 1816761 (UNILEVER) 24 July 1969 5ee page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON) 26 June 1956 5ee figures 1-3; claims 1,8						
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched?    III. DOCUMENTS CONSIDERED TO BE RELEVANT?   Category						
III. DOCUMENTS CONSIDERED TO BE RELEVANT®  Category® Citation of Document, 11 with Indication, where appropriate, of the relevant passages 12  X FR, A, 1600708 (SICALY) 4 September 1970 see page 2, lines 11-45; abstract, points 1-3  X DE, A, 1816761 (UNILEVER) 24 July 1969 see page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON) 26 June 1956 see figures 1-3; claims 1,8						
Citation of Document, "I with Indication, where appropriate, of the relevant passages 12   Relevant to Claim						
Category   Citation of Document, "I with indication, where appropriate, of the relevant passages 12   Relevant to Claim						
X FR, A, 1600708 (SICALY) 4 September 1970 see page 2, lines 11-45; abstract, points 1-3  X DE, A, 1816761 (UNILEVER) 24 July 1969 see page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON) 26 June 1956 see figures 1-3; claims 1,8	- 1/- 12					
## A September 1970   see page 2, lines 11-45; abstract, points 1-3    DE, A, 1816761 (UNILEVER)	n No. 13					
24 July 1969 see page 2, paragraph 4; page 3, paragraphs 2-3; page 4, paragraphs 1-3  X US, A, 2751687 (R.F. COLTON) 26 June 1956 see figures 1-3; claims 1,8	,15,					
26 June 1956 see figures 1-3; claims 1,8						
DE A. 1692705 (P. KÖLLN)						
16 March 1972	•					
A NL, A, 288628 (LEYBOLD) 10 March 1965						
A US, A, 3315619 (KELLOGG CO.) 25 April 1967 ./.	Y					
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the considered to be of particular relevance invention filing date.  "E" earlier document but published on or after the international filing date invention of the internation of the internation of the internation of the internation of the international filing date of the art.  "O" document referring to an oral disclosure, use, exhibition or other means.  "P" document published prior to the international filing date but later than the priority date claimed.  IV. CERTIFICATION  Date of the Actual Completion of the International Search  2nd October 1989  International Searching Authority  "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention.  "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered no						
	_					
EUROPEAN PATENT OFFICE T.K. WILLIS						

International Application No.

PCT/IT 89/00044

ategory *	Cita	tion of (	Document, with in	dication, where appro	ponate, of the relevant	passages	Relevant to Claim h
.						<u>,</u>	i
A	FR,	Ar	1395921	(LEYBOLD)	1965		1
. !					-		
i				· ·			
Î				-			
				•	*		
į	-						
•							
İ							
-						-	
İ					-		
-		-	-				
-							
				•			
						•	
- 9							
*		_		-			
							9
1							
!							
	•			٠			
; 							
	:			-			-
					•		-
Ì	,	•					
1							
.				* .		•	
					. •		
							aft entire ver
İ							
	•						

Form PCT ISA:210 (extra sheet) (January 1985)

# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

IT 8900044

29484 SA

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 30/10/89
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
FR-A- 1600708	27-07-70	None		
DE-A- 1816761	24-07-69	BE-A- FR-A- NL-A-	<b>72</b> 5952 1596364 <b>6</b> 717658	23-06-69 24-07-70 01-07-69
US-A- 2751687		None		
DE-A- 1692705	16-03-72	None		
NL-A- 288628		None		ny digo high man oan' 1000 1000 000 1000 1000 1000 1000
US-A- 3315619		None		
FR-A- 1395921		None		